## **CETIFICATION**

SDG No:

JC15796R

Laboratory:

Accutest, New Jersey

Site:

BMSMC, Building 5 Area

Matrix:

Groundwater

SM04.00.06 Humacao, PR

**SUMMARY:** 

Groundwater samples (Table 1) were collected on the BMSMC facility – Building 5 area. The BMSMC facility is located in Humacao, PR. Samples were taken March 7, 2016 and were analyzed in Accutest Laboratory of Dayton, New Jersey that reported the data under SDG No.: JC15796R. Results were validated using the latest guidelines (July, 2015) of the EPA Hazardous Waste Support Section. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. Data sample organic data samples summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

SAMPLE ID	SAMPLE DESCRIPTION	ANALYSIS PERFORMED
JC15796-1R	S-29R	TCL LIST PESTICIDES
JC15796-1RMS	S-9R2MS	TCL LIST PESTICIDES
JC15796-1RMSD	S-29RMSD	TCL LIST PESTICIDES
JC15796-2R	S-31R(2)	TCL LIST PESTICIDES
JC15796-3R	EB030716	TCL LIST PESTICIDES

Reviewer Name:

Rafael Infante

**Chemist License 1888** 

Signature:

Date:

May 5, 2016

A 1506600

#### **SGS Accutest**

# Report of Analysis

By

BP

Page 1 of 1

Client Sample ID: S-29R

File ID

4G66284R.D

Lab Sample ID: JC1

JC15796-1R AQ - Ground Water

Prep Date

03/13/16

Date Sampled: 03/07/16 Date Received: 03/09/16

Matrix: Method:

SW846 8081B SW846 3510C

Percent Solids: n/a

OP92024

Q

Project:

BMSMC, Building 5 Area, PR

Analyzed

03/20/16

Prep Batch An

Analytical Batch G4G1744

Run #1 Run #2

Initial Volume Final Volume

Run #1 1000 ml

10.0 ml

DF

Run #2

#### Pesticide TCL List

CAS No.	Compound	Result	RL	MDL	Units
309-00-2	Aldrin	ND	0.010	0.0060	ug/l
319-84-6	alpha-BHC	ND	0.010	0.0060	ug/l
319-85-7	beta-BHC	ND	0.010	0.0057	ug/l
319-86-8	delta-BHC	ND	0.010	0.0046	ug/l
58-89-9	gamma-BHC (Lindane)	ND	0.010	0.0028	ug/l
5103-71-9	alpha-Chlordane	ND	0.010	0.0046	ug/l
5103-74-2	gamma-Chlordane	ND	0.010	0.0046	ug/l
60-57-1	Dieldrin	ND	0.010	0.0036	ug/l
72-54-8	4,4'-DDD	ND	0.010	0.0038	ug/l
72-55-9	4,4'-DDE	ND	0.010	0.0062	ug/l
50-29-3	4,4'-DDT	ND	0.010	0.0050	ug/l
72-20-8	Endrin	ND	0.010	0.0050	ug/l
1031-07-8	Endosulfan sulfate	ND	0.010	0.0053	ug/l
7421-93-4	Endrin aldehyde	ND	0.010	0.0051	ug/l
53494-70-5	Endrin ketone	ND	0.010	0.0051	ug/l
959-98-8	Endosulfan-I	ND	0.010	0.0050	ug/l
33213-65-9	Endosulfan-II	ND	0.010	0.0043	ug/l
76-44-8	Heptachlor	ND	0.010	0.0038	ug/l
1024-57-3	Heptachlor epoxide	ND	0.010	0.0065	ug/l
72-43-5	Methoxychlor	ND	0.020	0.0057	ug/l
8001-35-2	Toxaphene	ND	0.25	0.18	ug/l
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limit	ts

2051-24-3 Decachlorobiphenyl 156% <sup>a</sup> 10-118%

(a) High percent recoveries and no positive found in the sample.



877-09-8

877-09-8

2051-24-3

123%

119%

170% a

Tetrachloro-m-xylene

Tetrachloro-m-xylene

Decachlorobiphenyl

26-132%

26-132%

10-118%

ND = Not detected

MDL = Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

#### SGS Accutest

# Report of Analysis

Ву

BP

03/13/16

Analyzed

03/20/16

Client Sample ID: S-31R(2) Lab Sample ID:

File ID

1000 ml

4G66285R.D

Matrix:

JC15796-2R AQ - Ground Water

DF

10.0 ml

1

Method: SW846 8081B SW846 3510C Project:

BMSMC, Building 5 Area, PR

G4G1744

Date Sampled: 03/07/16 Date Received: 03/09/16 Percent Solids: n/a

OP92024

Q

Prep Date Prep Batch **Analytical Batch** 

Run #1 Run #2

**Initial Volume** Final Volume

Run #1 Run #2

# Pesticide TCL List

CAS No.	Compound	Result	RL	MDL	Units
309-00-2	Aldrin	ND	0.010	0.0060	ug/l
319-84-6	alpha-BHC	ND	0.010	0.0060	ug/l
319-85-7	beta-BHC	ND	0.010	0.0057	ug/l
319-86-8	delta-BHC	ND	0.010	0.0046	ug/l
<b>58-89</b> -9	gamma-BHC (Lindane)	ND	0.010	0.0028	ug/l
5103-71-9	alpha-Chlordane	ND	0.010	0.0046	ug/l
5103-74-2	gamma-Chlordane	ND	0.010	0.0046	ug/l
60-57-1	Dieldrin	ND	0.010	0.0036	ug/l
72-54-8	4,4'-DDD	ND	0.010	0.0038	ug/l
72-55-9	4,4'-DDE	ND	0.010	0.0062	ug/l
50-29-3	4,4'-DDT	ND	0.010	0.0050	ug/I
72-20-8	Endrin	ND	0.010	0.0050	ug/l
1031-07-8	Endosulfan sulfate	ND	0.010	0.0053	ug/l
7421-93-4	Endrin aldehyde	ND	0.010	0.0051	ug/l
53494-70-5	Endrin ketone	ND	0.010	0.0051	ug/l
959-98-8	Endosulfan-I	ND	0.010	0.0050	ug/l
33213-65-9	Endosulfan-II	ND	0.010	0.0043	ug/l
76-44-8	Heptachlor	ND	0.010	0.0038	ug/l
1024-57-3	Heptachlor epoxide	ND	0.010	0.0065	ug/l
72-43-5	Methoxychlor	ND	0.020	0.0057	ug/l
8001-35-2	Toxaphene	ND	0.25	0.18	ug/l
CAS No.	Surrogate Recoveries	Run#1	Run# 2	Limi	ts
877-09-8	Tetrachloro-m-xylene	92%		26-13	32%

(a) High percent recoveries and no positive found in the sample.



877-09-8

2051-24-3

2051-24-3

105%

115%

122% a

Tetrachloro-m-xylene

Decachlorobiphenyl

Decachlorobiphenyl

26-132%

10-118%

10-118%

ND = Not detected RL = Reporting Limit

MDL = Method Detection Limit

**E** = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

# Report of Analysis

Page I of 1

Client Sample ID: EB030716

Lab Sample ID: JC15796-3R Matrix:

AQ - Equipment Blank Method: SW846 8081B SW846 3510C Date Sampled: 03/07/16 Date Received: 03/09/16 Percent Solids: n/a

Q

Project: BMSMC, Building 5 Area, PR

File ID DF Analyzed By Prep Date Prep Batch **Analytical Batch** Run #1 4G66286R.D 03/20/16 BP 03/13/16 OP92024 G4G1744

Run #2

Initial Volume Final Volume 1000 ml 10.0 ml

Run #1 Run #2

#### Pesticide TCL List

CAS No.	Compound	Result	RL	MDL	Units
309-00-2	Aldrin	ND	0.010	0.0060	ug/l
319-84-6	alpha-BHC	ND	0.010	0.0060	ug/l
319-85-7	beta-BHC	ND	0.010	0.0057	ug/l
319-86-8	delta-BHC	ND	0.010	0.0046	ug/l
58-89-9	gamma-BHC (Lindane)	ND	0.010	0.0028	ug/l
5103-71-9	alpha-Chlordane	ND	0.010	0.0046	ug/l
5103-74-2	gamma-Chlordane	ND	0.010	0.0046	ug/l
60-57-1	Dieldrin	ND	0.010	0.0036	ug/I
72-54-8	4,4'-DDD	ND	0.010	0.0038	ug/l
72-55-9	4,4'-DDE	ND	0.010	0.0062	ug/l
50-29-3	4,4 -DDT	ND	0.010	0.0050	ug/l
72-20-8	Endrin	ND	0.010	0.0050	ug/l
1031-07-8	Endosulfan sulfate	ND	0.010	0.0053	ug/l
7421-93-4	Endrin aldehyde	ND	0.010	0.0051	ug/l
53494-70-5	Endrin ketone	ND	0.010	0.0051	
959-98-8	Endosulfan-I	ND	0.010	0.0050	
33213-65-9	Endosulfan-II	ND	0.010	0.0043	
76-44-8	Heptachlor	ND	0.010	0.0038	_
1024-57-3	Heptachlor epoxide	ND	0.010	0.0065	
72-43-5	Methoxychlor	ND	0.020	0.0057	
8001-35-2	Toxaphene	ND	0.25	0.18	ug/l
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	ts
877-09-8	Tetrachloro-m-xylene	110%		26-13	32%
959-98-8 33213-65-9 76-44-8 1024-57-3 72-43-5 8001-35-2 CAS No.	Endosulfan-I Endosulfan-II Heptachlor Heptachlor epoxide Methoxychlor Toxaphene Surrogate Recoveries	ND ND ND ND ND ND ND	0.010 0.010 0.010 0.010 0.020 0.25	0.0050 0.0043 0.0038 0.0065 0.0057 0.18	ts



ND = Not detected

877-09-8

2051-24-3

2051-24-3

MDL = Method Detection Limit

116%

84%

89%

RL = Reporting Limit

E = Indicates value exceeds calibration range

Tetrachloro-m-xylene

Decachlorobiphenyl

Decachlorobiphenyl

J = Indicates an estimated value

26-132%

10-118%

10-118%

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound





Method: SW846 8081B

# Matrix Spike/Matrix Spike Duplicate Summary

Job Number: JC15796R

AMANYWP Anderson, Mulholland & Associates Account:

Project: BMSMC, Building 5 Area, PR

Sample         File ID           OP92024-MS         4G66282.1           OP92024-MSD         4G66283.1           JC15796-1R         4G66284.1           JC15796-1         4G66284.1	D 1	Analyzed 03/20/16 03/20/16 03/20/16 03/20/16	By BP BP BP	Prep Date 03/13/16 03/13/16 03/13/16 03/13/16	Prep Batch OP92024 OP92024 OP92024 OP92024	Analytical Batch G4G1744 G4G1744 G4G1744 G4G1744
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The QC reported here applies to the following samples:

JC15796-1R, JC15796-2R, JC15796-3R

CAS No.	Compound	JC15796-1 ug/l Q		MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
309-00-2	Aldrin	ND	0.25	0.31	124	0.25	0.35	140* a	12	37-159/40
319-84-6	alpha-BHC	ND	0.25	0.34	136	0.25	0.39	156* a	14	37-164/37
319-85-7	beta-BHC	ND	0.25	0.31	124	0.25	0.34	136	9	46-151/36
319-86-8	delta-BHC	ND	0.25	0.34	136	0.25	0.39	156* a	14	32-168/36
58-89-9	gamma-BHC (Lindane)	ND	0.25	0.33	132	0.25	0.38	152* *	14	44-160/37
510 <b>3-</b> 71-9	alpha-Chlordane	ND	0.25	0.38	152* a	0.25	0.44	176* a	15	38-160/35
5103-74-2	gamma-Chlordane	ND	0.25	0.40	160	0.25	0.45	180* a	12	39-157/37
60-57-1	Dieldrin	ND	0.25	0.35	140	0.25	0.41	164* a	16	42-161/36
72-54-8	4,4'-DDD	ND	0.25	0.35	140	0.25	0.41	164* a	16	40-161/36
72-55-9	4,4'-DDE	ND	0.25	0.35	140	0.25	0.41	164* a	16	34-158/36
50-29-3	4,4'-DDT	ND	0.25	0.41	164* a	0.25	0.48	192* a	16	41-173/33
72-20-8	Endrin	ND	0.25	0.40	164* B	0.25	0.47	188* a	16	44-166/35
1031-07-8	Endosulfan sulfate	ND	0.25	0.43	172* a	0.25	0.50	200* a	15	46-161/36
7421-93-4	Endrin aldehyde	ND	0.25	0.37	148* a	0.25	0.43	172* a	15	34-149/36
53494-70-5	Endrin ketone	ND	0.25	0.38	152* a	0.25	0.44	176* a	15	44-157/36
959-98-8	Endosulfan-I	ND	0.25	0.35	140* a	0.25	0.40	160* a	13	43-154/35
33213-65-9	Endosulfan-II	ND	0.25	0.37	148* a	0.25	0.43	172* a	15	40-162/35
76-44-8	Heptachlor	ND	0.25	0.32	128	0.25	0.37	148* a	14	33-153/37
1024-57-3	Heptachlor epoxide	ND	0.25	0.36	144* a	0.25	0.43	172* a	18	45-154/37
72-43-5	Methoxychlor	ND	0.25	0.49	196* a	0.25	0.58	232* a	17	48-169/32
8001-35-2	Toxaphene	ND		ND			ND		nc	50-150/30
CAS No.	Surrogate Recoveries	MS	MSD	JC:	1 <i>5</i> 796-1R	JC1579	6-1 Lin	nits		
877-09-8	Tetrachloro-m-xylene	106%	126%* *	123	1%	123%	26-	132%		
877-09-8	Tetrachloro-m-xylene	103%	123%* ª			119%		132%		
0051 04 0	-									

180%\* a

167%\* a

170%\* b

156%\* b

170%\* b

156%\* b

10-118%

10-118% SOCIA

(a)	Outside	the	OC	limits.

2051-24-3 Decachlorobiphenyl

2051-24-3 Decachlorobiphenyl

(b) High percent recoveries and no positive found in the sample.

147%\* a

138%\* a

<sup>\* -</sup> Outside of Control Limits.

Anderson Multipolised & Associates  Anderson Multipolised & Associates  Anderson Multipolised & Associates  Purchase NY 10577  City State 21p  Torry Taylor Sand Napart to:  Phone 6: 914-251-0400	Project Name  Location  ProjectPO a:  BMS: Building & Arms	Accessed John F. CURS 5.713  Accessed John F. CURS 5.713  Accessed Garden F. CURS 5.713  Accessed Garden F. CURS 5.713  Accessed Garden F. CURS 5.713	
3 EBO30116 37/16	Time   Sampled   Manny   Follow   Proservation		
21 Day Standard   Approved by   14 Day   7 Days State Fraction   15 Days   15 Days	Commercial "A"    Na Full   Commercial "B"   Fill CIP   ASP Category B   Dies Deliverable   State Forms   Other (Specify)   decumented before each time semples change posteocion, including forms of the companion of the companio	Federal Express ID # 6012 Y 53 5170 Lab Trip Blank Date 4/37//6 Time 1000 SVOC Analysis by Method 8270D SIM for 1/4 Dioxane and Naphthalene only.  Dispring 133 Procedure By:    Document   Procedure   Procedure   Procedure	

JC15796R: Chain of Custody Page 1 of 4

#### **EXECUTIVE NARRATIVE**

SDG No:

JC15796R

Laboratory:

**Accutest, New Jersey** 

Analysis:

SW846-8081B

Number of Samples:

5

Location:

BMSMC, Building 5 Area

Humacao, PR

SUMMARY:

Five (5) groundwater samples were analyzed for selected pesticides following method SW846-8081B. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence *Hazardous Waste Support Section SOP No. HW-36A, Revision O, June, 2015. SOM02.2. Pesticide Data Validation.* The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** 

None

Major:

None

Minor:

- 1. Several surrogate recoveries (tetrachloro-m-xylene and decachlorobiphenyl) outside laboratory control limits in sample JC15796-1R; -2R; Blank Spike; JC15796-1RMS and -1RMSD. No action taken, surrogate recoveries within control limits in at least one of the columns except for the Blank Spike sample (QC sample).
- 2. Several analytes MS/MSD recoveries outside the laboratory/guidance validation document criteria. Percent recoveries high and analytes not detected in the sample. No action taken, professional judgment.
- 3. Florisil and GPC cartridge performance check data not included in data package. No action taken.

**Critical findings:** 

None

Major findings:

None

**Minor findings:** 

None

COMMENTS:

Results are valid and can be used for decision making purposes.

Reviewers Name:

Rafael Infante

Chemist License 1888

Signature:

Date:

May 5, 2016

# SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: JC15796-1R

Sample location: BMSMC Building 5 Area

Sampling date: 7-Mar-16
Matrix: Groundwater

METHOD: 8081B

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Aldrin	0.010	ug/L	1	-	U	Yes
alpha-BHC	0.010	ug/L	1	- 2	U	Yes
beta-BHC	0.010	ug/L	1	100	U	Yes
delta-BHC	0.010	ug/L	1	12	U	Yes
gamma-BHC (Lindane)	0.010	ug/L	1	-	U	Yes
alpha-Chlordane	0.010	ug/L	1	12	U	Yes
gamma-Chlordane	0.010	ug/L	1		U	Yes
Dieldrin	0.010	ug/L	1	1,2	U	Yes
4,4'-DDD	0.010	ug/L	1	17.	U	Yes
4,4'-DDE	0.010	ug/L	1	2	U	Yes
4,4'-DDT	0.010	ug/L	1		U	Yes
Endrin	0.010	ug/L	1	2	U	Yes
Endosulfan sulfate	0.010	ug/L	1		U	Yes
Endrin aldehyde	0.010	ug/L	1	2	U	Yes
Endrin ketone	0.010	ug/L	1	-5	U	Yes
Endosulfan-I	0.010	ug/L	1	2	U	Yes
Endosulfan-II	0.010	ug/L	1		U	Yes
Heptachlor	0.010	ug/L	1	-	U	Yes
Heptachlor epoxide	0.010	ug/L	1	5	U	Yes
Methoxychlor	0.020	ug/L	1	2	U	Yes
Toxaphene	0.25	ug/L	1	-	U	Yes

Sample ID: JC15796-2R

Sample location: BMSMC Building 5 Area

Sampling date: 7-Mar-16 Matrix: Groundwater

## METHOD: 8081B

Analyte Name	Result	Units	<b>Dilution Factor</b>	Lab Flag	Validation	Reportable
Aldrin	0.010	ug/L	1	-	U	Yes
alpha-BHC	0.010	ug/L	1	-	U	Yes
beta-BHC	0.010	ug/L	1	-	U	Yes
delta-BHC	0.010	ug/L	1	-	U	Yes
gamma-BHC (Lindane)	0.010	ug/L	1	-	U	Yes
alpha-Chlordane	0.010	ug/L	1	-	U	Yes
gamma-Chlordane	0.010	ug/L	1	-	U	Yes
Dieldrin	0.010	ug/L	1	-	U	Yes
4,4'-DDD	0.010	ug/L	1	-	U	Yes
4,4'-DDE	0.010	ug/L	1	-	U	Yes
4,4'-DDT	0.010	ug/L	1	-	U	Yes
Endrin	0.010	ug/L	1	-	U	Yes
Endosulfan sulfate	0.010	ug/L	1	-	U	Yes
Endrin aldehyde	0.010	ug/L	1	-	U	Yes
Endrin ketone	0.010	ug/L	1	-	U	Yes
Endosulfan-I	0.010	ug/L	1	-	U	Yes
Endosulfan-II	0.010	ug/L	1	-	U	Yes
Heptachlor	0.010	ug/L	1	•	U	Yes
Heptachlor epoxide	0.010	ug/L	1	-	U	Yes
Methoxychlor	0.020	ug/L	1	-	U	Yes
Toxaphene	0.25	ug/L	1	-	U	Yes

Sample ID: JC15796-3R

Sample location: BMSMC Building 5 Area

Sampling date: 7-Mar-16 Matrix: Groundwater

### METHOD: 8081B

Analyte Name	Result	Units	<b>Dilution Factor</b>	Lab Flag	Validation	Reportable
Aldrin	0.011	ug/L	1	-	U	Yes
alpha-BHC	0.011	ug/L	1	-	U	Yes
beta-BHC	0.011	ug/L	1	-	U	Yes
delta-BHC	0.011	ug/L	1	-	U	Yes
gamma-BHC (Lindane)	0.011	ug/L	1	-	U	Yes
alpha-Chlordane	0.011	ug/L	1	-	ប	Yes
gamma-Chlordane	0.011	ug/L	1	-	U	Yes
Dieldrin	0.011	ug/L	1	-	ប	Yes
4,4'-DDD	0.011	ug/L	1	-	U	Yes
4,4'-DDE	0.011	ug/L	1	-	U	Yes
4,4'-DDT	0.011	ug/L	1	-	U	Yes
Endrin	0.011	ug/L	1	-	U	Yes
Endosulfan sulfate	0.011	ug/L	1	-	U	Yes
Endrin aldehyde	0.011	ug/L	1	-	U	Yes
Endrin ketone	0.011	ug/L	1	-	U	Yes
Endosulfan-I	0.011	ug/L	1	-	U	Yes
Endosulfan-II	0.011	ug/L	1	-	U	Yes
Heptachlor	0.011	ug/L	1	-	U	Yes
Heptachlor epoxide	0.011	ug/L	1	-	U	Yes
Methoxychlor	0.022	ug/L	1	-	U	Yes
Toxaphene	0.28	ug/L	1	-	U	Yes

	Sampling Date:March_7,_2016
	Shipping Date:March_8,_2016 EPA Region No.:2
REVIEW OF PESTICIDE C	DRGANIC PACKAGE
The following guidelines for evaluating volar required validation actions. This document will judgment to make more informed decision and users. The sample results were assessed according order of precedence HW-36A, Revision 0, June, 2015. SOM02.2. Pest data validation actions listed on the data reguidance document, unless otherwise noted.	assist the reviewer in using professional din better serving the needs of the data ording to USEPA data validation guidance to Hazardous Waste Support Section SOP No. Sicide Data Validation. The QC criteria and
The hardcopied (laboratory name) _Accutest	data package received has been mmarized. The data review for VOCs included:
Lab. Project/SDG No.:JC15796R No. of Samples:5_	Sample matrix: _Groundwater
Trip blank No.:	
X Data CompletenessX Holding TimesN/A GC/MS TuningX Internal Standard PerformanceX BlanksX Surrogate RecoveriesX Matrix Spike/Matrix Spike Duplicate  Overall Comments:Pesticides_TCL_list_by_SW844	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Definition of Qualifiers:  J- Estimated results  U- Compound not detected  R- Rejected data  UJ- Estimated nondetect	
Reviewer:Rafuel Defaut Date:May_5,_2016	

# **DATA REVIEW WORKSHEETS**

# DATA COMPLETENESS

MISSING INFORMATION	DATE LAB, CONTACTED	DATE RECEIVED
4		
		5075.00%
_		
N.		
	1	<u> </u>
	1	
z		
	- W	
		<u></u>
	-A - 27 - 425	
	6795	

All criteria were met _X
Criteria were not met
and/or see below

#### **HOLDING TIMES**

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED/ANALYZED	ACTION

Preservatives:	_All_samples_extracted_and_analyzed_within_the_required_criteria	

#### Criteria

Aqueous samples - seven (7) days from sample collection for extraction; 40 days from sample collection for analysis.

Non-aqueous samples – fourteen (14) days from sample collection for extraction; 40 days from sample collection for analysis.

Cooler temperature (Criteria: 4 + 2 °C): 4.4°C - OK

#### **Actions**

# Qualify aqueous sample results using preservation and technical holding time information as follows:

- a. If there is no evidence that the samples were properly preserved ( $T = 4^{\circ}C \pm 2^{\circ}C$ ), and the samples were extracted or analyzed within the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ).
- b. If there is no evidence that the samples were properly preserved (T =  $4^{\circ}$ C  $\pm$   $2^{\circ}$ C), and the samples were extracted or analyzed outside the technical holding times, qualify detects as estimated (UJ).
- c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times, no qualification of the data is necessary.
- d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

- e. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
- f. If technical holding times are grossly exceeded, use professional judgment to qualify the data.

# Qualify non-aqueous sample results using preservation and technical holding time information as follows:

- a. If there is no evidence that the samples were properly preserved (T =  $4^{\circ}$ C  $\pm$   $2^{\circ}$ C), and the samples were extracted or analyzed within the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ).
- b. If there is no evidence that the samples were properly preserved (T =  $4^{\circ}$ C  $\pm$   $2^{\circ}$ C), and the samples were extracted or analyzed outside the technical holding time, qualify detects as estimated (UJ).
- c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time, no qualification of the data is necessary.
- d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.
- e. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
- f. If technical holding times are grossly exceeded, use professional judgment to qualify the data.

	All criteria were metX
Criteria	were not met see below

GAS CHROMATOGRAPH WITH ELECTRON CAPTURE DETECTOR (GC/ECD) INSTRUMENT PERFORMANCE CHECK (SECTIONS 1 TO 5)

#### 1. Resolution Check Mixture

#### Criteria

Is the resolution between two adjacent peaks in the Resolution Check Mixture C greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column?

Yes? or No?

is the resolution between two adjacent peaks in the Resolution Check Mixture (A and B) greater than or equal to 60.0%?

Yes? or No?

Note: If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if

coelution exists.

#### Action

- a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).

# 2. Performance Evaluation Mixture (PEM) Resolution Criteria

#### Criteria

Is PEM analysis performed at the required frequency (at the end of each pesticide initial calibration sequence and every 12 hours)?

Yes? or No?

#### Action

a. If PEM is not performed at the required frequency, qualify all associated sample and blank results as unusable (R).

#### Criteria

Is PEM % Resolution < 90%?

Yes? or No?

#### Action

- a. a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).

	All criteria were metX	
Criteria	were not met see below	

## 3. PEM 4,4'-DDT Breakdown

Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected?

Yes? or No?

Action

a. Qualify detects for 4,4'-DDT; detects for 4,4'-DDD; and detects for 4,4'-DDE as estimated (J)

Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected

Yes? or No?

Action

- a. Qualify non-detects for 4,4'- DDT as unusable (R)
- b. Qualify detects for 4,4'-DDD as tentatively identified (NJ)
- c. Qualify detects for 4,4'-DDE as tentatively identified (NJ)

#### 4. PEM Endrin Breakdown

Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is detected?

Yes? or No?

Action

a. Qualify detects for Endrin; detects for Endrin aldehyde; and detects for Endrin ketone as estimated (J)

Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is not detected

Yes? or No?

Action

- a. Qualify non-detects for Endrin as unusable (R)
- b. Qualify detects for Endrin aldehyde as tentatively identified (NJ)
- c. Qualify detects for Endrin ketone as tentatively identified (NJ)

All criteria were metX	
Criteria were not met see below	

# 5. Mid-point Individual Standard Mixture Resolution -

#### Criteria

Is the resolution between two adjacent peaks in the Resolution Check Mixture C greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column?

Yes? or No?

Is the resolution between two adjacent peaks in the Resolution Check Mixture (A and B) greater than or equal to 90.0%?

Yes? or No?

Note: If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if coelution exists.

#### Action

- a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).

#### Criteria

Is mid-point individual standard mixture analysis performed at the required frequency (every 12 hours)?

Yes? or No?

### Action

a. If the mid-point individual standard mixture analysis is not performed at the required frequency, qualify all associated sample and blank results as unusable (R).

All criteria were metX
Criteria were not met
and/or see below

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	03/18/16	
Dates of continuing calibr	ation:03/18/16_(initial);_03/20/16;_03/21/16	<u>`</u>
Instrument ID numbers:_	GC4G	
Matrix/Level:	Aqueous/low	

DATE	LAB FILE ID#	CRITERIA OUT RFs, %RSD, <u>%</u> <b>D</b> , r	COMPOUND	SAMPLES AFFECTED
03/20/16	cc1741-50	-64.1 %/-31.8	Methoxychlor	JC15796-1R; -2R; and -3R

Note: Initial and continuing calibration meets the required criteria except for the cases described above. Results for Metoxychlor qualified as estimated (UJ) in affected samples. Closing calibration performed and within the required criteria.

#### Criteria

Are a five point calibration curve delivered with concentration levels as shown in Table 3 of SOP HW-36A, Revision 0, June, 2015?

Yes? or No?

## **Actions**

If the standard concentrations listed in Table 3 are not used, use professional judgment to evaluate the effect on the data

#### Criteria

Are RT Windows calculated correctly?

Yes? or No?

#### Action

Recalculate the windows and use the corrected values for all evaluations.

#### Criteria

Are the Percent Relative Standard Deviation (%RSD) of the CFs for each of the single component target compounds less than or equal to 20.0%, except for alpha-BHC and delta-BHC?

Yes? or No?

#### **DATA REVIEW WORKSHEETS**

Are the %RSD of the CFs for alpha-BHC and delta-BHC less than or equal to 25.0%. Yes? or No?

Is the %RSD of the CFs for each of the Toxaphene peaks must be < 30% when 5-point ICAL is performed?

Yes? or No?

Is the %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) less than or equal to 30.0%.

Yes? or No?

#### Action

- a. If the %RSD criteria are not met, qualify detects as estimated (J) and use professional judgment to qualify non-detected target compounds.
- b. If the %RSD criteria are within allowable limits, no qualification of the data is necessary

## **Continuing Calibration Checks**

#### Criteria

Is the continuing calibration standard analyzed at the acceptable time intervals? Yes? or No?

#### Action

- a. If more than 14 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either a PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C), qualify all data as unusable (R).
- b. If more than 12 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence, qualify all data as unusable (R).
- c. If more than 72 hours has elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3), qualify all data as unusable (R).

#### Criteria

Is the Percent Difference (%D) within ±25.0% for the PEM sample?

Yes? or No?

#### Action

a. Qualify associated detects as estimated (J) and non-detects as estimated (UJ).

#### Criteria

For the Calibration Verification Standard (CS3); is the Percent Difference (%D) within ±25.0%?

Yes? or No?

#### Action

Qualify associated detects as estimated (J) and non-detects as estimated (UJ).

#### DATA REVIEW WORKSHEETS

All criteria were met \_\_X\_\_ Criteria were not met and/or see below \_\_\_\_\_

#### Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected?

Yes? or No?

#### Action

- a. Qualify detects for 4,4'-DDT; detects for 4,4'-DDD; and detects for 4,4'-DDE as estimated (J)
- b. Non-detected associated compounds are not qualified

#### Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected

Yes? or No?

#### Action

- a. Qualify non-detects for 4,4'- DDT as unusable (R)
- b. Qualify detects for 4,4'-DDD as tentatively identified (NJ)
- c. Qualify detects for 4,4'-DDE as tentatively identified (NJ)

#### Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is detected?

Yes? or No?

#### Action

- a. Qualify detects for Endrin; detects for Endrin aldehyde; and detects for Endrin ketone as estimated (J)
- b. Non-detected associated compounds are not qualified

#### Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is not detected

Yes? or No?

#### Action

- a. Qualify non-detects for Endrin as unusable (R)
- b. Qualify detects for Endrin aldehyde as tentatively identified (NJ)
- c. Qualify detects for Endrin ketone as tentatively identified (NJ)

A separate worksheet should be filled for each initial curve

All criteria were met _X
Criteria were not met
and/or see below

# BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contami	nation in the bla	anks below. Hig	gh and low levels blanks	must be treated separately.
CRQL concentra	ationN	/A	· · · · · · · · · · · · · · · · · · ·	
Laboratory blank	ks			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
			•	it_of_0.01_and_0.001_ug/L.
	-			
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
			olankNo_field/trip_blan	k_analyzed_with_this_data_

All criteria were met _X
Criteria were not met
and/or see below

### BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

The concentration of non-target compounds in all blanks must be less than or equal to 10  $\mu$ g/L. The concentration of each target compound found in the method or field blanks must be less than its CRQL listed in the method.

Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

Specific actions are as follows:

# **Blank Actions for Pesticide Analyses**

Blank Type	Blank Result	Sample Result	Action for Samples
	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥CRQL	No qualification required
Method, Sulfur		< CRQL	Report CRQL value with a U
Cleanup,		≥ CRQL and ≤ blank	Report blank value for
Instrument, Field,	> CRQL	concentration	sample concentration with a
TCLP/SPLP			U
		≥ CRQL and > blank	No qualification required
		concentration	
1	= CRQL	≤CRQL	Report CRQL value with a U
		> CRQL	No qualification required
	Gross contamination	Detects	Report blank value for
			sample concentration with a
			U

All criteria were met _X
Criteria were not met
and/or see below

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
		+			
		-			
				<del>                                     </del>	

All criteria were met
Criteria were not met
and/or see belowX

#### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix:_Ground	lwater				
Lab Sample ID	Lab File ID	Sl a	Sl b	S2 a	S2 b
JC15796-1R JC15796-2R JC15796-3R OP92024-BS1 OP92024-MB1 OP92024-MS OP92024-MSD	4G66284.D 4G66285.D 4G66286.D 4G66281.D 4G66280.D 4G66282.D 4G66283.D	123 92 110 107 139* d 106 126* c	119 105 116 112 138* d 103 123* e	170* c 115 84 129* d 158* d 147* c 180* e	156* c 122* c 89 133* d 154* d 138* e 167* e
Surrogate Compounds S1 = Tetrachloro-m-xylene S2 = Decachlorobiphenyl		Recover Limits 26-1329 10-1189	- /o		

- (a) Recovery from GC signal #1
- (b) Recovery from GC signal #2
- (c) High percent recoveries and no positive found in the sample.
- (d) High percent recoveries and no positive found in the QC batch.
- (e) Outside the OC limits.

**Note:** No action taken, surrogate recoveries within the required criteria (> 30 % - < 150 %) in at least one of the columns.

#### Actions:

- a. For any surrogate recovery greater than 150%, qualify detected target compounds as biased high (J+).
- b. Do not qualify non-detected target compounds for surrogate recovery > 150 %.
- c. If both surrogate recoveries are greater than or equal to 30% and less than or equal to 150%, no qualification of the data is necessary.
- d. For any surrogate recovery greater than or equal to 10% and less than 30%, qualify detected target compounds as biased low (J-).
- e. For any surrogate recovery greater than or equal to 10% and less than 30%, qualify non-detected target compounds as approximated (UJ).

## **DATA REVIEW WORKSHEETS**

- f. If low surrogate recoveries are from sample dilution, professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor:
  - i. Qualify detected target compounds as biased low (J-).
  - ii. Qualify non-detected target compounds as unusable (R).
- g. If surrogate RTs in PEMs, Individual Standard Mixtures, samples, and blanks are outside of the RT Windows, the reviewer must use professional judgment to qualify data.
- h. If surrogate RTs are within RT windows, no qualification of the data is necessary.
- i. If the two surrogates were not added to all samples, MS/MSDs, standards, LCSs, and blanks, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

# **Summary Surrogate Actions for Pesticide Analyses**

	Action*			
Criteria	Detected Target	Non-detected Target		
	Compounds	Compounds		
%R > 150%	J+	No qualification		
30% < %R < 150%	No qualification			
10% < %R < 30%	J-	UJ		
%R < 10% (sample dilution not a factor)	J-	R		
%R < 10% (sample dilution is a factor)	Use professional judgment			
RT out of RT window	Use professional judgment			
RT within RT window	No qualification			

<sup>\*</sup> Use professional judgment in qualifying data, as surrogate recovery problems may not directly apply to target analytes.

All criteria were met
Criteria were not met
and/or see belowX

# MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

## 1. MS/MSD Recoveries and Precision Criteria

Data for MS and MSDs will not be present unless requested by the Region.

Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD, unless designated as such by the Region.

NOTE: For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

Note: No qualification of sample results, professional judgment.

Action

A separate worksheet should be used for each MS/MSD pair.

## **DATA REVIEW WORKSHEET**

# JC15796R - MS/MSD

Compound	JC15796 ug/l	i-1R Q	Spike ug/l	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
Aldrin alpha-BHC delta-BHC gamma-BHC (Lindane) alpha-Chlordane gamma-Chlordane Dieldrin 4,4'-DDD 4,4'-DDE 4,4'-DDT Endrin Endosulfan sulfate Endrin aldehyde Endrin ketone Endosulfan-I Endosulfan-II	ND ND ND ND ND ND ND ND ND ND ND ND ND N	Q	0.25 0.25 0.25 0.25 0.25 0.25 0.25 0.25	0.31 0.34 0.34 0.33 0.38 0.40 0.35 0.35 0.35 0.41 0.40 0.43 0.37 0.38 0.35	124 136 136 132 152* a 160 140 140 140 164* a 164* a 172* a 148* a 140* a 148* a	0.25 0.25 0.25 0.25 0.25 0.25 0.25 0.25	0.35 0.39 0.39 0.38 0.44 0.45 0.41 0.41 0.41 0.48 0.47 0.50 0.43 0.44 0.40 0.43	140° a 156° a 156° a 152° a 176° a 180° a 164° a 164° a 192° a 188° a 200° a 172° a 160° a 172° a	12 14 14 15 12 16 16 16 16 15 15 15	37-159/40 37-164/37 32-168/36 44-160/37 38-160/35 39-157/37 42-161/36 40-161/36 34-158/36 41-173/33 44-166/35 46-161/36 34-149/36 44-157/36 43-154/35 40-162/35
Heptachlor Heptachlor epoxide Methoxychlor Toxaphene	ND ND ND ND		0.25 0.25 0.25	0.32 0.36 0.49 ND	128 144* a 196* a	0.25 0.25 0.25	0.37 0.43 0.58 ND	148* a 172* a 232* a	14 18 17 nc	33-153/37 45-154/37 48-169/32 50-150/30
Surrogate Recoveries Tetrachloro-m-xylene Tetrachloro-m-xylene Decachlorobiphenyl Decachlorobiphenyl	MS 106% 103% 147%* a 138%* a	MSD 126%* a 123%* a 180%* a 167%* a	119% 170%* b		JC15796 123% 119% 170%* b 156%* b		Limits 26-132% 26-132% 10-118% 10-118%			

<sup>(</sup>a) Outside the QC limits.

<sup>(</sup>b) High percent recoveries and no positive found in the sample.

All criteria were met _X
Criteria were not met
and/or see below

# LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

### 1. LCS Recoveries Criteria

LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50 – 120
Heptachlor epoxide	50 – 150
Dieldrin	30 – 130
4,4'-DDE	50 – 150
Endrin	50 – 120
Endosulfan sulfate	50 – 120
trans-Chlordane	30 – 130
Tetrachloro-m-xylene (surrogate)	30 – 150
Decachlorobiphenyl (surrogate)	30 – 150

LCS	concentrations:	0.25_ug/L		
List the %R	of compounds wh	ich do not meet the criteria	n	
	LCS ID	COMPOUND	% R	QC LIMIT

#### Action

The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

- a. If the LCS recovery exceeds the upper acceptance limit, qualify detected target compounds as estimated (J). Do not qualify non-detected target compounds.
- b. If the LCS recovery is less than the lower acceptance limit, qualify detected target compounds as estimated (J) and non-detects as unusable (R).
- c. Use professional judgment to qualify data for compounds other than those compounds that are included in the LCS.
- d. Use professional judgment to qualify non-LCS compounds. Take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in the performance of the LCS compound to the non-LCS compound.
- e. If the LCS recovery is within allowable limits, no qualification of the data is necessary.

### **DATA REVIEW WORKSHEETS**

# 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? <u>Yes</u> or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

All criteria were met
Criteria were not met
and/or see belowN/A

#### FLORISIL CARTRIDGE PERFORMANCE CHECK

NOTE: Florisil cartridge cleanup is mandatory for all extracts.

#### Criteria

Is the Florisil cartridge performance check conducted at least once on each lot of cartridges used for sample cleanup or every 6 months, whichever is most frequent?

Yes? or No?

N/A

#### Criteria

Are the results for the Florisil Cartridge Performance Check solution included with the data package?

Yes? or No?

N/A

Note: If % criteria are not met, examine the raw data for the presence of polar interferences and use professional judgment in qualifying the data as follows:

#### Action:

- a. If the Percent Recovery is greater than 120% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.
- b. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
- c. If the Percent Recovery is greater than or equal to 10% and less than 80% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected target compounds as estimated (JJ) and non-detected target compounds as approximated (UJ).
- d. If the Percent Recovery is less than 10% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J) and qualify non-detected target compounds as unusable (R).
- e. If the Percent Recovery of 2,4,5-trichlorophenol in the Florisil Cartridge Performance Check is greater than or equal to 5%, use professional judgment to qualify detected and non-detected target compounds, considering interference on the sample chromatogram.

Note: State in the Data Review Narrative potential effects on the sample data resulting from the Florisil Cartridge Performance Check analysis not yielding acceptable results.

Note: No information for florisil cartridge performance check included in data package. No qualification of the data performed, professional judgment.

All criteria were met				
Criteria were not met				
and/or see belowN/A				

### GEL PERMEATION CHROMATOGRAPHY (GPC) PERFORMANCE CHECK

NOTE: GPC cleanup is mandatory for all soil samples.

If GPC criteria are not met, examine the raw data for the presence of high molecular weight contaminants; examine subsequent sample data for unusual peaks; and use professional judgment in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory chooses to analyze samples under unacceptable GPC criteria.

#### Action:

- a. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, the non-detected target compounds may be suspect, qualify detected compounds as estimated (J).
- b. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, qualify all non-detected target compounds as unusable (R).
- c. If the Percent Recovery is greater than or equal to 10% and is less than 80% for any of the pesticide target compounds in the GPC calibration, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
- d. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
- e. If high recoveries (i.e., greater than 120%) were obtained for the pesticides and surrogates during the GPC calibration check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.

Note: State in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

Note:\_ No information for performance of GPC cleanup included in data package. No qualification of the data performed, professional judgment.

All criteria were metX
Criteria were not met
and/or see below

#### TARGET COMPOUND IDENTIFICATION

#### Criteria:

- 1. Is Retention Times (RTs) of both of the surrogates and reported target compounds in each sample within the calculated RT Windows on both columns?

  Yes? or No?
- 2. Is the Tetrachloro-m-xylene (TCX) RT ±0.05 minutes of the Mean RT (RT) determined from the initial calibration and Decachlorobiphenyl (DCB) within ±0.10 minutes of the RT determined from the initial calibration?

  Yes? or No?
- 3. Is the Percent Difference (%D) for the detected mean concentrations of a pesticide target compound between the two Gas Chromatograph (GC) columns within the inclusive range of  $\pm$  25.0 %?

  Yes? or No?
- 4. When no analytes are identified in a sample; are the chromatograms from the analyses of the sample extract and the low-point standard of the initial calibration associated with those analyses on the same scaling factor?

  Yes? or No?
- 5. Does the chromatograms display the Single Component Pesticides (SCPs) detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale.

  Yes? or No?
- 6. If an extract is diluted; does the chromatogram display SCPs peaks between 10-100% of full scale, and multi-component analytes between 25-100% of full scale?

  Yes? or No?

  N/A
- 7. For any sample; does the baseline of the chromatogram return to below 50% of full scale before the elution time of alpha-BHC, and also return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of DCB?

  Yes? or No?
- 8. If a chromatogram is replotted electronically to meet these requirements; is the scaling factor used displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram submitted in the data package.

  Yes? or No?

#### Action:

- a. If the qualitative criteria for both columns were not met, all target compounds that are reported as detected should be considered non-detected.
- b. Use professional judgment to assign an appropriate quantitation limit using the following guidance:
  - If the detected target compound peak was sufficiently outside the pesticide RT Window, the reported values may be a false positive and should be replaced with the sample Contract Required Quantitation Limits (CRQL) value.

- ii. If the detected target compound peak poses an interference with potential detection of another target peak, the reported value should be considered and qualified as unusable (R).
- c. If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate RT Windows, but was reported as a non-detect, the compound may be a false negative. Use professional judgment to decide if the compound should be included.

Note: State in the Data Review Narrative all conclusions made regarding target compound identification.

- d. If the Toxaphene peak RT windows determined from the calibration overlap with SCPs or chromatographic interferences, use professional judgment to qualify the data.
- e. If target compounds were detected on both GC columns, and the Percent Difference between the two results is greater than 25.0%, consider the potential for coelution and use professional judgment to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, use professional judgment to determine how best to report, and if necessary, qualify the data according to these guidelines.
- f. If Toxaphene exhibits a marginal pattern-matching quality, use professional judgment to establish whether the differences are due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of Toxaphene is strongly suggested, report results as presumptively present (N).

# GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) CONFIRMATION

NOTE: This confirmation is not usually provided by the laboratory. In cases where it is provided, use professional judgment to determine if data qualified with "C" can be salvaged if it was previously qualified as unusable (R).

#### Action:

- a. If the quantitative criteria for both columns were met ( $\geq 5.0$  ng/µL for SCPs and  $\geq 125$  ng/µL for Toxaphene), determine whether GC/MS confirmation was performed. If it was performed, qualify the data using the following guidance:
  - i. If GC/MS confirmation was not required because the quantitative criteria for both columns was not met, but it was still performed, use professional judgment when evaluating the data to decide whether the detect should be qualified with "C".
  - ii. If GC/MS confirmation was performed, but unsuccessful for a target compound detected by GC/ECD analysis, qualify those detects as "X".

All criteria were metX					
Criteria were not met					
and/or see below					

# COMPOUND QUANTITATION AND REPORTED CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

BLANK SPIKE

beta-BHC

RF = 0.596

 $[] = (71879704)(50)/(215.9X10^{6})(0.596)$ 

= 27.93 ppb Ok

#### Action:

- a. If sample quantitation is different from the reported value, qualify result as unusable (R).
- b. When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample.
- c. Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original reporting form and substituting the data from the diluted sample.
- d. Results between the MDL and CRQL should be qualified as estimated (J).
- e. Results less than the MDL should be reported at the CRQL and qualified (U). MDLs themselves are not reported.
- f. For non-aqueous samples, if the percent moisture is less than 70.0%, no qualification of the data is necessary. If the percent moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent moisture is greater than or equal to 90.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table).

# Percent Moisture Actions for Pesticide Analysis for Non-Aqueous Samples

Criteria	Action		
	Detected Associated Compounds	Non-detected Associated Compounds	
% Moisture < 70.0	No qualification		
70.0 < % Moisture < 90.0	J	UJ	
% Moisture > 90.0	J	R	

#### DATA REVIEW WORKSHEETS

ist samples which h	ave ≤ 50 % solids		

lote: If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

# Dilution performed

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
,		
		<del>-</del>
	<del></del>	

All criteria were metN/A
Criteria were not met
and/or see below

#### FIELD DUPLICATE PRECISION

NOTE: In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples. Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. If large RPDs (> 50%) is observed, confirm identification of samples and note difference in the executive summary.

Sample IDs	: <u> </u>	<del>-</del>		Matrix:	<del>-</del>
COMPOUND	SQL ug/L	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laboratory of precision. RPD with	lata incluing the the	lded with this of quired criteria of	lata package. MS/MSD of < 50 %.	% recovery	RPD used to assess
		<u> </u>			1

#### Actions:

- a. Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.
- b. If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:
  - i. If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).
  - ii. If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.
  - iii. If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.
  - iv. If both sample and duplicate results are not detected, no action is needed.

#### OVERALL ASSESSMENT OF DATA

### Action:

- 1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
- 2. Write a brief narrative to give the user an indication of the analytical limitations of the data.

Note: The Contract Laboratory Program Project Officer (CLP PO) must be informed if any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

Overall assessment of the data: Results are valid; the data can be used for

decision making purposes.